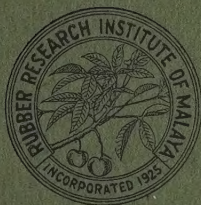


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VARIATION IN THE COMPOSITION AND PROPERTIES OF FRESH LATEX AND SUBSEQUENT CHANGES ON AMMONIATION *

BY

EDGAR RHODES

Summary

Systematic examination has been made on 163 samples of latex drawn at the approximate rate of three per week over a period of fourteen months from a single tapper's task on old seedling trees, newly opened after several years' rest on the continuous alternate daily system. The latex samples were all examined, both in the fresh condition and after 10 days and 15 days preservation with 0.7% ammonia; during the last eight months certain tests were also made on the latex immediately after ammoniation on the day of collection.

The tests included dry rubber content, total solids, acetone extract, and acid number of whole-latex rubber films, surface tension, viscosity, stirring stability, and ease of creaming.

It has been observed that:—

(i) The difference between percentage total solids and percentage dry rubber content, is greater after 10 days preservation than in the fresh condition.

(ii) The acetone extract and its acid number from whole-latex film are less in freshly-ammoniated latex than in fresh latex, and very much greater in ammoniated latex stored for 10 days. The acid number after 10 days preservation is usually about twice that of the fresh latex. These findings and their implications are discussed in the text in which experimental figures are given. High dry rubber content usually connotes low acetone extract and acid value in the fresh latex.

(iii) Values for surface tension before and after ammoniation are given. The fall in surface tension as a result of ammoniation has not been proven to be governed by the changes in acetone soluble material, or the acid number thereof.

(iv) The viscosity of ammoniated latex is shown to be very significantly correlated with its dry rubber content. In the case of fresh latex the degree of correlation is much less; a possible reason for this is advanced. Experimental results are given.

* This paper is substantially the same as that published in the *India Rubber Journal*, Volume 97 (1939) 82.

(v) The initial stirring stability is shown to be influenced by the initial dry rubber content of the latex. The data do not provide an adequate explanation of the changes which take place in stability on storage.

(vi) The creaming ease of new latex appears also to be influenced by its initial dry rubber content, but the present data are not adequate fully to explain the variations which are noted.

Introduction

This paper describes results which have been obtained on latex from a small area of old seedling rubber close to the Rubber Research Institute of Malaya, and under close supervision by it, over a period of fourteen months from the time of opening after having been out of tapping for some years. It represents an attempt to follow some of the changes in composition and properties of latex, which are of special interest to those concerned more with preserved and concentrated latex as an article of commerce, than variations in the properties of crude rubber. For this reason changes in dry-rubber-content, total solids, acetone, extract, and acid value of whole latex film, surface tension, viscosity and stability have all been followed both in the fresh latex and the preserved latex made from it.

Roberts (1938) also studied in the laboratories of the Rubber Research Institute the changes which occur in ammoniated latex using the method of analysis developed by him (1936).

Experimental

The latex used in the experiments came from a single tapper's task in a four-acre block of rubber trees situated within fifty yards of the Rubber Research Institute at Kuala Lumpur. The trees were brought into regular alternate-daily tapping on 1st April, 1937, after having been rested for some years, and latex was examined regularly from that date until the end of May, 1938. Because the area is so close to the laboratories, supervision is easy and the fresh latex can be brought in for examination much more quickly and much earlier than on the normal rubber estate. The possibilities of error, owing to changes caused by incipient putrefaction in the field, are thus reduced to a minimum.

In the course of the fourteen months' period, 163 samples of latex were drawn for examination from the alternate-daily tappings. This represents nearly three samples per week.

When each sample was drawn, it was divided into two parts. The one part was examined immediately in the unammoniated condition; the other part was ammoniated to 0.7 per cent ammonia

(with a 25 per cent solution of ammonia in water), and on this ammoniated part some tests were made immediately, and others after storage periods of 10 days and 50 days respectively.

The following table shows the tests which were made on the various samples:—

TABLE I

Test	Fresh on first day	Immediately after ammoniation first day	10 days after ammonia- tion	50 days after ammonia- tion
Dry rubber content ...	Yes	—	Yes	—
Total solids ...	Yes	—	Yes	—
Acetone extract ...	Yes	Yes*	Yes	Yes
Acid number ...	Yes	Yes*	Yes	Yes
Surface tension ...	Yes	—	Yes	Yes
Viscosity ...	Yes	—	Yes	Yes
Stirring stability ...	—	—	Yes	Yes
Ease of creaming ...	Yes	—	—	—

* Acetone extract and acid numbers were determined on the freshly ammoniated samples during the last eight months only.

The procedure for the various tests was as under:—

(i) *Dry rubber content*.—Determined by acid coagulation of a known weight of latex (about 20 gm.). The coagulum is rolled thin with ample washing during rolling, dried at 70° C., and weighed.

(ii) *Total solids*.—A known weight of latex (about 10 gm.) is evaporated to dryness at 70°C. in a shallow weighed vessel, and the weight of dry solids determined.

(iii) *Acetone extract*.—About 30 cc. of latex, whether fresh or ammoniated, are spread very thinly on a glass frame (21 in. square). Air is blown over the film by an electric fan. After two hours the dried whole-latex rubber is removed, cut into small pellets and stored overnight in a desiccator. Five grammes of the whole-latex rubber are then extracted for eight hours with acetone. The weight of acetone-soluble matter is determined after removal of the solvent.

(iv) *Acid number*.—The whole extract from each sample is dissolved in 20 cc. alcohol (95 per cent), and titrated with N/100 alkali against phenolphthalein as indicator. The acid value is expressed as the number of mgm. KOH required to neutralize the extract from 100 gm. whole-latex rubber.

(v) *Surface tension*.—Determined with a Du Nüoy Tensiometer of the direct reading type. Surface skinning is avoided by placing the vessel containing the latex inside a deeper dish, and covering this loosely with two flat pieces of glass, which leave only sufficient opening to admit the wire carrying the loop.

(vi) *Viscosity*.—Determined with a Höppler Viscosimeter at 30° C.

(vii) *Stirring stability*.—Determined with the slowspeed apparatus devised by the London Advisory Committee for Rubber Research. Latex is diluted to a dry rubber content of 30 per cent, and after adjusting the ammonia content to 0.7 per cent, 600 cc. are stirred at 600 r.p.m. in presence of 162 gm. zinc oxide (90 per cent calculated on dry rubber or 27 per cent calculated on the volume of latex), which is blown in dry during the first 30 seconds of stirring. Stirring proceeds to the "viscosity end point" described by Rhodes (1936).

(viii) *Ease of creaming*.—200 cc. latex are mixed with 14 cc. of 3 per cent Tragon A solution (0.2 per cent Tragon A on the latex), and left for creaming. After a period of 48 hours the serum is removed and the dry rubber content of the cream is determined. This dry rubber content is taken as the index of "ease of creaming."

It will be observed that stirring stability tests were not made on the freshly ammoniated latex. The reason is that because of the slowness of the stirring and the very high results always given by very fresh latex, it was not possible to complete a test in a working day.

The individual alternate-daily records are not given in this paper because of their bulk. Instead, in Table II the monthly means for the various properties are shown. Each of the figures given is, therefore, the mean for approximately twelve samples per month. Graphs of these monthly means form Figures 1 to 8 inclusive.

In Table III are given correlation coefficients for various pairs of properties. These were calculated from the actual individual day-to-day records, and not from the monthly means.

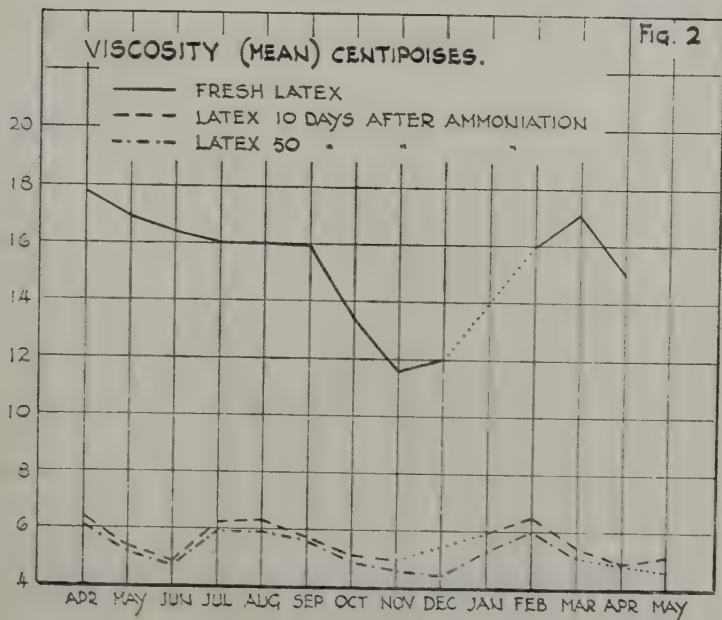
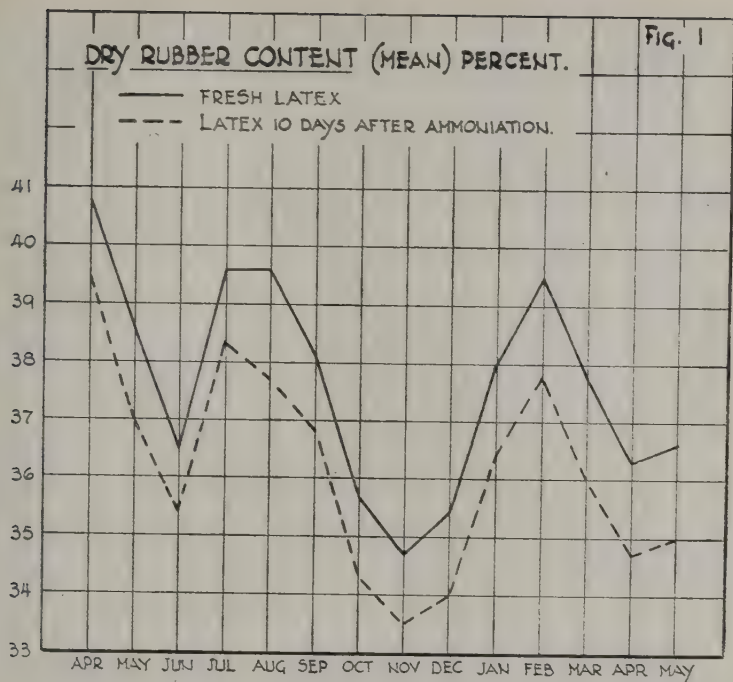
Monthly Means			Dry rubber content and total solids					
			Fresh Latex before ammoniation			After 10 days ammoniation		
Month			DRC %	TS %	non rubbers	DRC %	TS %	non rubbers
April	1937	...	40.8	43.5	2.7	39.5	42.6	3.1
May	1937	...	38.6	41.0	2.4	37.0	39.9	2.9
June	1937	...	36.5	39.0	2.5	35.4	38.2	2.8
July	1937	...	39.6	42.1	2.5	38.3	41.2	2.9
Aug.	1937	...	39.6	42.0	2.4	37.7	40.8	3.1
Sept.	1937	...	38.1	40.5	2.4	36.8	39.7	2.9
Oct.	1937	...	35.7	38.2	2.5	34.3	37.2	2.9
Nov.	1937	...	34.7	37.3	2.6	33.5	36.6	3.1
Dec.	1937	...	35.4	38.1	2.7	34.0	36.9	2.9
Jan.	1938	...	38.0	40.4	2.4	36.4	39.4	3.0
Feb.	1938	...	39.5	42.0	2.5	37.8	40.9	3.1
Mar.	1938	...	37.8	40.1	2.3	36.0	38.9	2.9
April	1938	...	36.3	38.7	2.4	34.7	37.7	3.0
May	1938	...	36.6	38.9	2.3	35.0	38.0	3.0

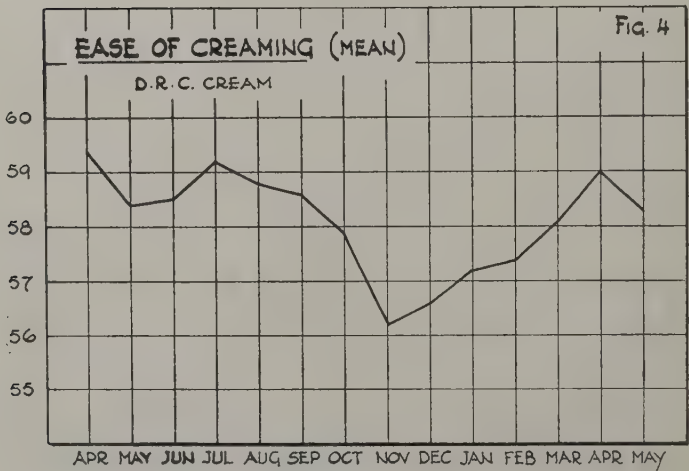
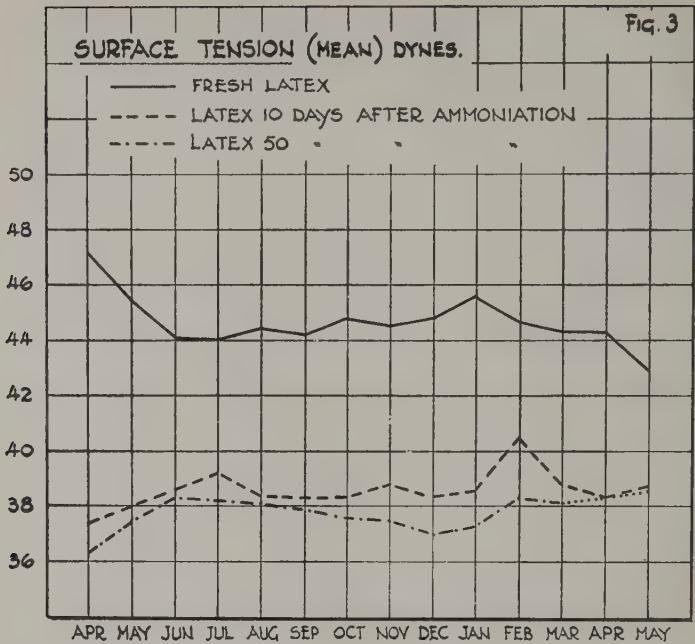
TABLE II

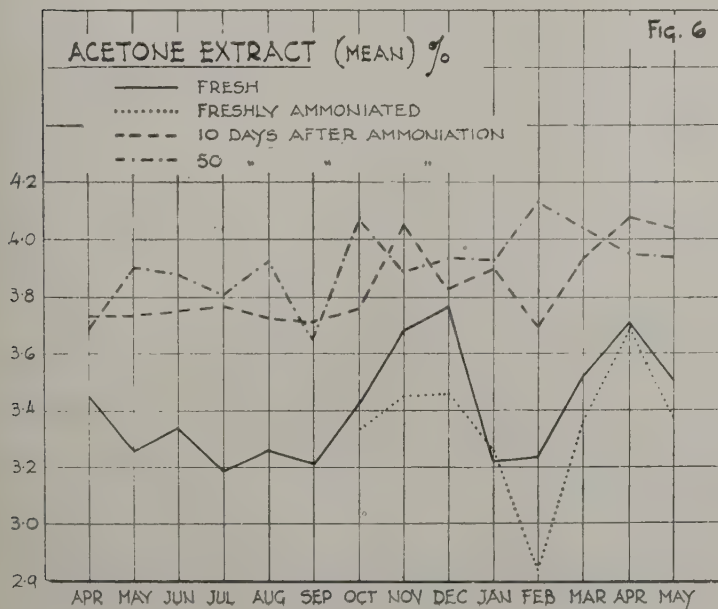
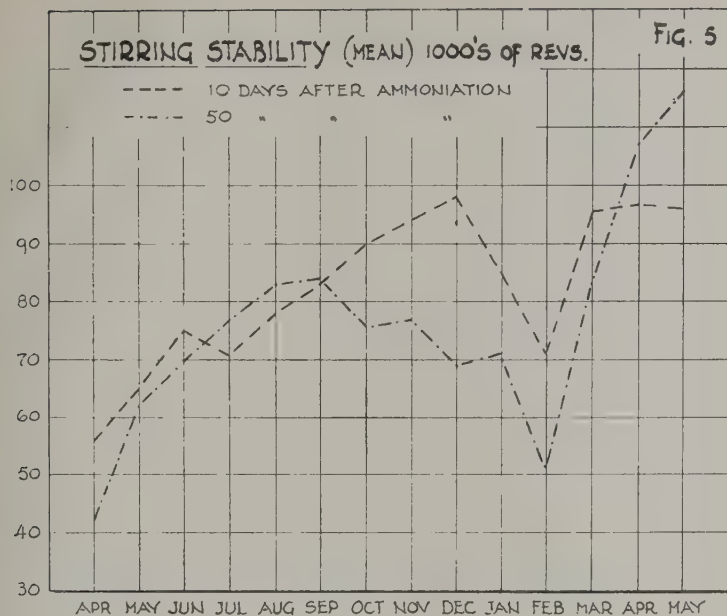
Monthly Means for various Latex Characteristics

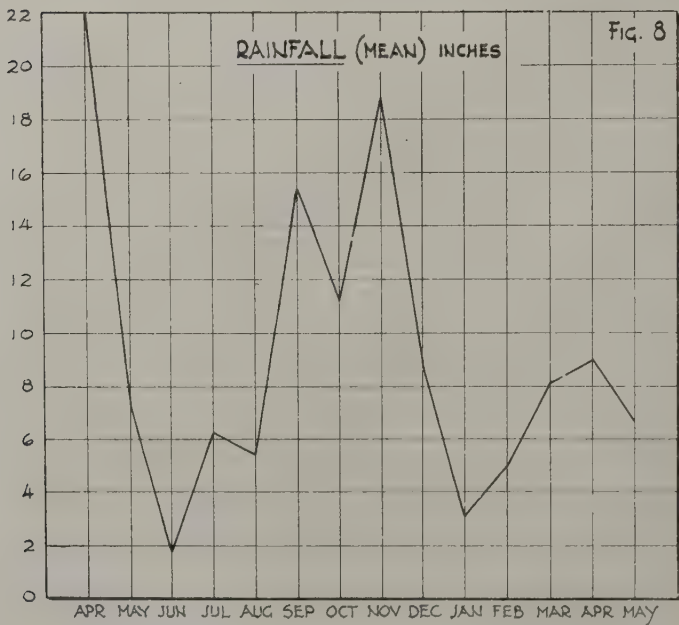
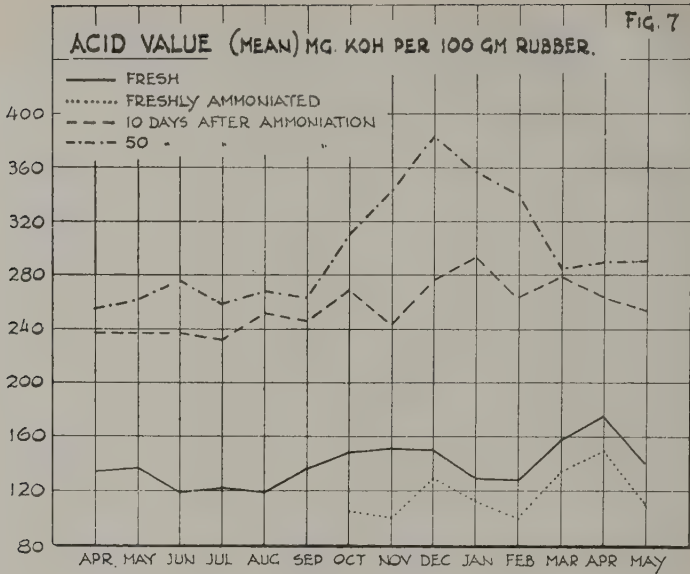
Acetone Extract %				Acid number of Acetone extract (mgm. KOH per 100 gm. rubber)				Surfa
esh	Freshly ammoni- ated	After 10 days ammoni- ation	After 50 days ammoni- ation	Fresh	Freshly ammoni- ated	After 10 days ammoni- ation	After 50 days ammoni- ation	Fresh
5	—	3.74	3.68	133	—	237	254	47.2
6	—	3.74	3.90	136	—	237	262	45.5
14	—	3.75	3.88	119	—	237	276	44.1
9	—	3.77	3.81	123	—	231	258	44.0
16	—	3.73	3.93	119	—	252	268	44.4
11	—	3.71	3.65	136	—	247	264	44.2
2	3.33	3.76	4.07	148	106	269	309	44.8
8	3.45	4.06	3.89	152	101	243	343	44.6
7	3.46	3.83	3.94	150	129	276	386	44.8
12	3.25	3.90	3.93	128	113	293	361	45.6
14	2.93	3.69	4.13	127	101	264	340	44.7
2	3.36	3.94	4.04	158	133	279	286	44.3
12	3.69	4.08	3.95	176	149	264	289	44.3
10	3.38	4.04	3.94	141	111	254	290	42.9

Viscosity (dynes)		Viscosity (centipoises)			Stirring stability. (27% ZnO 600 r.p.m. in 1000's of revs.)		Ease of creaming as DRC cream after 48 hour period
	50 days after ammoniation	Fresh	10 days after ammoniation	50 days after ammoniation	10 days after ammoniation	50 days after ammoniation	
4	36.3	17.8	6.4	6.1	56	42	59.4
9	37.5	16.9	5.4	5.2	65	62	58.4
5	38.3	16.4	4.8	4.7	75	70	58.5
2	38.2	16.0	6.2	5.9	71	77	59.2
4	38.1	16.0	6.3	5.9	78	83	58.8
6	37.9	15.9	5.7	5.6	83	84	58.6
	37.6	13.4	5.1	4.9	90	76	57.9
	37.5	11.6	4.9	4.6	94	77	56.2
	37.0	12.0	—	4.4	98	69	56.6
	37.3	—	5.9	5.3	85	71	57.2
	38.3	16.0	6.5	6.0	71	51	57.4
	38.1	17.1	5.5	5.1	96	83	58.1
	—	15.0	4.9	—	97	107	59.0
	38.6	—	5.1	4.6	96	116	58.3









Discussion

(a) GENERAL OBSERVATIONS

(i) *Dry rubber content and total solids.*—It will be seen from Figs. 1 and 8 that after the first three months, when the trees had reached full flow, the dry rubber content of the fresh latex was generally low in the wet months, and *vice versa*. This is as might have been expected.

Too much significance may not be attached to the fact that the values for dry rubber content after 10 days' storage are invariably lower than those for the fresh latex, because the difference must be due, mainly, to the dilution caused by the initial ammoniation with 25 per cent aqueous ammonia.

There is one feature worthy of note which arises from an examination of the monthly means for total solids and dry content of the fresh and the preserved samples. It will be observed from Table II that in the case of the fresh latex, the difference between total solids and dry rubber content varied from month to month between the limits of 2.3 and 2.7 per cent whereas in the case of the ammoniated latex the month-to-month differences were invariably higher, and lay between the limits of 2.8 and 3.1 per cent. It would appear, therefore, that ammonia has reacted with some part of the total solids in such a way as to render it non-precipitable by acid on coagulation for the determination of "dry rubber content."

(ii) *Acetone extract and acid number.*—It will be seen from Table II and from Figs. 6 and 7 that ammoniation produces striking changes in the amount of acetone-soluble bodies in the whole-latex rubber, and also in the acidity of the acetone-soluble matter.

During every month the acetone extract was greater after 10 days ammoniation than when fresh. In most months there was a slight further increase between 10 and 50 days.

In the case of the acid number, the differences were still more striking. After 10 days' ammoniation, the acid number of the acetone extract was approximately double that of the fresh latex, and further increases appear invariably to occur between 10 and 50 days. In some months these further increases were very large.

A further interesting feature is provided by the values for acetone extracts and acid values for whole rubber prepared from latex immediately after ammoniation on the day of collection. It would be expected that the figures for such rubbers would be indistinguishable from those for the fresh unammoniated latex. This is not, however, the case. Table II and Figs. 6 and 7 give the

monthly means for freshly-ammoniated latex for eight months, and it will be seen that during six of those months, the acetone extract means for fresh latex were the higher, while during only two months were they virtually the same as those for the freshly-ammoniated material. More marked differences are shown by the eight monthly means for the acid numbers of the acetone extract from the fresh and freshly-ammoniated material. Here it is seen that the acid number of the whole rubber from the freshly-ammoniated latex was invariably less than that from the fresh latex rubber. The only explanation of this unexpected finding which can be advanced at present is that "fresh" latex usually contains, besides the normal acetone solubles, a certain small amount of adventitious acetone soluble acid produced by the action of bacteria between tapping and collection, and that the addition of ammonia produces from the bacterial acid a salt which is insoluble in acetone. This would account for the lower acetone extract and acid number of freshly-ammoniated latex, but it is emphasized that there is at present no practical proof of the correctness of such a hypothesis.

These values for the changes in acetone extract and acid number of whole-latex rubber from fresh, freshly-ammoniated and stored latex are quite the most worthy of note of any in this paper. They indicate first a reduction in acetone extract and acid number on addition of ammonia, followed by a progressive hydrolysis presumably by ammonia, which is usually well-advanced in ten days, and thereafter proceeds more slowly. It is not possible to deduce from the available figures whether or not the changes are complete in fifty days, but it seems probable that they are very nearly so. The results demonstrate a tangible difference between the composition of freshly-ammoniated latex, and the preserved latex of commerce.

Roberts (*loc. cit.*) also found that the "fatty acid complex" of fresh latex, composed of a considerable number of alcoholic and acidic substances in chemical combination, is extensively hydrolysed under the conditions of ammoniation, and is isolable as a corresponding ligroin-soluble mixture of the individual constituents of the original complex viz. alcohols, fatty acids, glucoside and terpenic material.

The view is commonly held that the acid number of whole latex rubber results from free fatty and other acids, but that in ammoniated latex these acids are present as ammonium soaps and salts. If this view is correct then these ammonium soaps, etc., must decompose again to free acids either during the drying of the latex film at room temperature, or during the process of acetone extraction, otherwise it is difficult to see in what manner the acid number of the acetone extract of whole-latex rubber could double

TABLE III
Correlation of Properties

Property	Correlated with	No. of Pairs	Coefficient of Correlation	Significance* (SS, S, or —)
D.R.C. Fresh latex	(i) Acid number. Fresh latex	135	-.282	SS
	(ii) Acetone extract. Fresh latex	135	-.390	SS
	(iii) Ease of creaming. Fresh latex	127	-.394	SS
	(iv) Viscosity. Fresh latex	82	.263	S
	(v) Stirring stability after 10 days' storage	134	-.369	SS
D.R.C. After 10 days' storage	(i) Acid number after 10 days' storage	151	-.169	S
	(ii) Acetone extract after 10 days' storage	151	-.340	SS
	(iii) Viscosity after 10 days' storage	122	.863	SS
	(i) Acid number. Fresh latex	146	.598	SS
Acetone extract. Fresh latex	(ii) Ease of creaming. Fresh latex	136	-.170	S
	(iii) Surface tension. Fresh latex	93	-.037	—
	(i) Acid number after 10 days' storage	156	.284	SS
Acetone extract after 10 days' storage	(ii) Surface tension after 10 days' storage	129	-.012	—
	(i) Acid number after 50 days' storage	159	.408	SS
Acetone extract after 50 days' storage	(ii) Surface tension after 50 days' storage	113	.129	—
	(i) Surface tension. Fresh latex	93	-.043	—
Acid number. Fresh latex	(ii) Ease of creaming. Fresh latex	136	-.0515	—
Acid number. After 10 days' storage	(i) Surface tension after 10 days' storage	129	-.324	SS
Acid number. After 50 days' storage	(i) Surface tension after 50 days' storage	113	-.135	—
Stirring stability. After 10 days' storage	(i) Acetone extract after 10 days' storage	155	.245	SS
	(ii) Acid number after 10 days' storage	155	.294	SS
Stirring stability. After 50 days' storage	(i) Acetone extract after 50 days' storage	158	-.110	—
	(ii) Acid number after 50 days' storage	158	-.242	SS

* SS = P < .01. Usually taken as highly significant.

S = P < .05. Usually taken as significant because there is between 1%—5% chance of the result having occurred by chance.

— = P > .05. Usually taken as not significant.

itself during the first ten days after preservation. The fact that the acid number of the acetone extract of freshly-ammoniated latex is less than that of the original fresh latex, lends colour to the view that the ammonia might have formed some soaps or salts and so reduced the acid value, but these lower results could obviously not have been obtained if decomposition had occurred again during drying and extraction. The evidence, therefore, leads to the speculation that the fatty acids may perhaps be present as such and not as soaps in ammoniated latex, as well as in fresh latex.

(iii) *Surface tension*.—From Table II and Fig. 3 it is seen that ammoniation and storage results in a fall in surface tension. The monthly means for fresh latex varied between 47.2 and 42.9 dynes. During 10 of the 14 months they lay between 44 and 45 dynes. The means for ammoniated latex 10 days old lay between 37.4 and 40.5 dynes, while those for latex 50 days old were always very slightly lower, and lay between 36.3 and 38.6 dynes.

Observations for which experimental figures are not presented here show that the fall in surface tension on ammoniation is not immediate as, say, in the case of viscosity. It progresses steadily at a rate of less than one dyne per 24 hours over the first ten days. This would indicate a gradual release of surface active substances by hydrolysis.

(iv) *Viscosity*.—The values in Table II and Fig. 2 illustrate the already well-known changes which take place when latex is ammoniated. Whereas the viscosity means for fresh latex between the dry rubber contents of 34.7 and 40.8 per cent show values between 11.6 and 17.8 centipoises, those for the ammoniated latex between 33.5 and 39.5 per cent dry-rubber lie in the zone 4.8-6.5 centipoises after 10 days' storage.

It will be observed from Figs. 1 and 2 that the curves for dry rubber content and viscosity after 10 days' storage, show maxima and minima in the same places. This is not strictly true in the case of viscosity of fresh latex versus dry rubber content. Fresh latex is, however, so prone to show exaggerated viscosity variations, due, perhaps, chiefly to incipient coalescence caused by bacterial infection, that this is not surprising.

(v) *Stirring stability*.—Table II and Fig. 5 indicate that during nine of the fourteen months the mean values for stability as measured by this technique, indicate a fall during the 10-50 day storage period, while during the other five months the reverse was the case. For this anomalous behaviour no explanation suggests itself.

From Figs. 1 and 5, however, there would appear to be some connection between dry rubber content and stirring stability after

ten days' storage, since the two relevant graphs are similar but reversed, showing maxima against minima, and vice versa, as if a rather low initial dry rubber content might connote a relatively high stability.

(vi) *Ease of creaming*.—If one omits from consideration the mean values for the months March to May, 1938, inclusive, Figs. 1 and 4 would seem to indicate a connection between dry rubber content and ease of creaming.

(b) CORRELATION OF PROPERTIES WITH ONE ANOTHER

In comparing various sets of means in Table II and in examining Figs. 1 to 8, sundry points of similarity have been seen. In order, however, to avoid faulty generalizations, Table III was prepared; this gives correlations of the actual day-to-day results for various pairs of properties, and enables one to form more accurate conclusions as to the dependence or otherwise of one factor on another. The relevant correlations are discussed below:—

(i) *Dry rubber content with acetone extract, and with acid number*.—From Table III the dry rubber content of fresh latex gives significant negative correlations with both the acetone extract and the acid number of whole-latex rubber from such latex. The results are derived from 135 samples of latex, and whole-rubber therefrom. Similarly, the correlations for dry rubber content in 10 days' old latex with the acetone extract and acid number are also negative and significant. Here the results are derived from 151 latex samples and rubbers. In no case, however, are the correlation coefficients very high, but it seems fairly safe to argue that low acetone extracts and acid numbers are to be suspected in the whole-latex rubbers from rubber-rich natural latices. If Fig. 1 is reversed it shows maxima and minima in approximately the same places as the relevant parts of Figs. 6 and 7.

(ii) *Acetone extract with acid number*.—There is positive and significant correlation between these two characters in the case of fresh latex, and also in latex stored for 10 and 50 days respectively. Since, however, the acetone extract itself contains the acids from which the acid number is derived, it is dangerous to draw conclusions from this data. The correlation may have no more meaning than that of the population of a town with the number of cases of whooping cough. It can, however, be said that high acid numbers are likely to be found when acetone extracts are high.

(iii) *Surface tension with acetone extract, and with acid numbers*.—The data in Table III show no significant correlation

between the surface tension of fresh latex and either the acetone extract or the acid number of the whole-latex rubber from it. In latex stored for 10 days there appears to be a negative and significant correlation between surface tension and acid number, but there is no correlation with acetone extract. After 50 days' storage no significant correlation is shown either with acetone extract or acid number.

It seems, therefore, that although the acids present in the acetone extract may at some stages during storage play some part in causing the fall in surface tension, those data provide no evidence that changes in the acetone extract and its acid number are the major factor in bringing about surface tension changes.

(iv) *Viscosity with dry rubber content.*—From Table III it is seen that the degree of correlation between the dry rubber content and the viscosity of fresh latex is not very high, whereas that between the dry rubber content and viscosity of the same latices after ammoniation and storage for 10 days is positive, significant, and very high, being of the order of 0.86.

This high value leaves no doubt that dry rubber content is the controlling factor in the viscosity of the preserved latex. It also adds weight to the view that one of the effects of ammonia on fresh latex is to disperse incipient aggregations which occur in fresh latex between tapping and collection, and which are responsible for the irregular viscosity values and consequent poor correlation with dry rubber content.

(v) *Stirring stability with dry rubber content, and with acetone extract and acid number.*—It is seen from Table III that the initial dry rubber content is negatively and significantly correlated with the stirring stability after 10 days' storage. This implies that a low initial dry rubber content usually connotes a rather high stability during the early stages of storage. Since under the present conditions of test all latex samples are diluted to the same dry rubber content (30 per cent) before testing, this would suggest that substances accessory to the rubber may play an important part in determining early stability. It has already been noted that acetone extract and acid number are correlated negatively with dry rubber content, and this leads to the possibility that the acetone extract or its acid number might be positively and significantly correlated with stirring stability. Examination of the data for acetone extract and acid number after 10 days' storage, with stability after 10 days' storage, shows that these characteristics are so correlated, but the coefficient of correlation is not very high. If now stability at 50 days' storage is set against acetone extracts and acid numbers for the same storage period, no correlation is

found with acetone extract, and a significant and negative one with acid number.

It is clear, therefore, that the available data do not provide an explanation of the changes in stability which occur on ammoniation and storage. The data indicate that low initial dry rubber content may tend to promote higher earlier stability, but there is no evidence that either it or the acetone extract and acid number are major factors in determining initial stability or subsequent changes in it.

(vi) *Ease of Creaming with dry rubber content, with acetone extract, and with acid number.*—The data in Table III show a positive and significant correlation between the initial dry rubber content of latex and its ease of creaming with Tragon A. Negative correlation also occurs with the acetone extract and the acid number of the fresh latex, but in the first case the correlation is not highly significant, and in the second it is without significance.

It is obvious, therefore, that although high initial dry rubber content may be a favourable factor in creaming, there are other factors probably more important which the present data are inadequate to disclose.

Acknowledgments

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